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DEVELOPMENT OF EMBEDDED SYSTEM BASED COLORIMETER FOR THE ANALYSIS OF NICKEL: AN ALGORITHMIC APPROACH

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ABSTRACT

The hardware and software of an embedded system based colorimeter, has developed, built and tested. It has designed to detect and measure automatically the contents of nickel in water. The light dependant resistor (LDR) and high flux light emitting diode (LED) were used as detector and source of light respectively. A constant current LED driver was constructed with the use of two transistors. The effect of dark current of LDR was compensated by use of hardwired potential divider network. The microcontroller PIC18F4550 was used to develop embedded system for the control, measurement, and analysis of absorption of light. An algorithm and C- language program have been written for the purpose of adjustment of zero absorption for the no-analyte reference, measurement of transmitted light intensity and its conversion into absorbance, detection of characteristics wavelength, calculation of absorptivity coefficient from the standard analyte solutions, and finally estimation of unknown concentration of nickel. The colorimeter is portable, easy to use, and it can be used for the detection of other metallic elements.

KEYWORDS: Colorimeter, LED, LDR, Microcontroller

1. INTRODUCTION

Nickel is a mineral and it is found in several food materials including nuts, grains and beans. Nickel alloys are characterized by strength, and resistance to corrosion and heat at high temperatures and for this reason it is used in gas turbines and rocket engines [1]. Nickel is released into the air by the industries manufacturing such products. It then falls down after reaction with raindrops. Some nickel compounds will adsorb to soil particles and become immobile as a result. Vegetables grown in soil contains small amount of nickel. Nickel uptake will boost when animals eat vegetables from polluted soils. For animals nickel is an essential mineral in small amount, but it can be hazardous when the maximum tolerable amount is exceeded. This can cause various kinds of diseases on internal organs of animals [2]. So, it is necessary to measure the concentration level of nickel in polluted soil and waste water. Colorimetric technique is one of the instrumental method for the analysis of metal particle in waste water [3, 4].

Modern colorimeter is constructed from the electronic devices such as light emitting diode (LED) [5] and photoconductive cell [6]. The colorimeter gives precise and stable result with the use of steady source of light, obtained from the constant current driven high flux LEDs. The accuracy depends on the resolution of analog to digital convertor (ADC) and data types used in microcontroller (μC). The various manual operations involved in the colorimeter can be automated by use of μC. The complete experiment can be automated by writing the software program. The procedure of particular analysis can be set by making necessary changes in the software.

In the present work, a prototype colorimeter has been developed for the analysis of nickel. In the developed colorimeter, modern electronic components are used for the construction of photoelectric sensor and automation section. A pair of semiconducting LED and photoconductive LDR forms the sensor, and highly integrated embedded PIC18F4550 microcontroller is used to automate the manual operations. The developed instrument can be used in the analytical chemistry laboratory for the conduct of experiments based on the colorimeter. The rest of the article is organized as follows: Section 2 provides the method of colorimetric analysis based on Beer-Lambert law. The circuit diagram is described in section 3, followed by Software section in 4. The results are discussed in Section 5. Finally, conclusions along with limitations and some of our thoughts for future scope are presented in section 6.

2. METHOD

Software program has been written to control the intensity of LED used as a source of light for the reference and sample solutions. The intensity of the incident light, I_0, for the reference solution is measured in terms of the analog voltage, Vr, by the microcontroller. Similarly, intensity of transmitted light, I, from sample solution is measured in terms of analog voltage, Vs. The software program measures the transmittance and calculates the absorbance with the use of Beer-Lambert law [7].

The fraction of I_0 that gets through the sample is called transmittance (T) is given by,

$$T = \frac{I}{I_0} \tag{1}$$

If the reference solution is blank such as distilled water then the total incident light is transmitted by the solution.

Then, $I=I_0$ and $T=1$.

However, solutions other than distilled water absorb some amount of incident light, and due to this transmitted intensity is less than incident one, i.e. $I<I_0$ and $T< 1$.

Direct measurement of absorbance is not possible due to the limitations in the colorimeter. But, it can be calculated from the transmittance.

$$A = - \log_{10} T,\ A = \log_{10} \left(\frac{1}{T}\right) = \log_{10}\left(\frac{I_0}{I}\right) \tag{2}$$

T is also given by, $T = 10^{-\alpha LC}$

Where: α = absorptivity coefficient, L= path length of light beam (cm); C = concentration of absorbing species.

$\log_{10}T = \log_{10} (10^{-\alpha LC}) = -\alpha L C$

$$A = - \log_{10}T = \alpha LC \tag{3}$$

In order to achieve the most accurate results of the concentration of colored species in solution, there is need to maximize the light absorbance by the species. Maximum absorbance can be obtained by varying the wavelength of light. The wavelength corresponding to the maximum absorbance, A_{max}, is called the characteristics wavelength (λ_{max}). Software program detects the type of chemical species from the database of λ_{max} and A_{max}. In automated colorimeter, the program automatically scans the wavelengths and determines absorbance. The graphical analysis can be carried out to visualize the wavelength response as shown in Figure 1. From the plot the wavelength corresponding to maximum absorbance (λ_{max}) can be obtained.

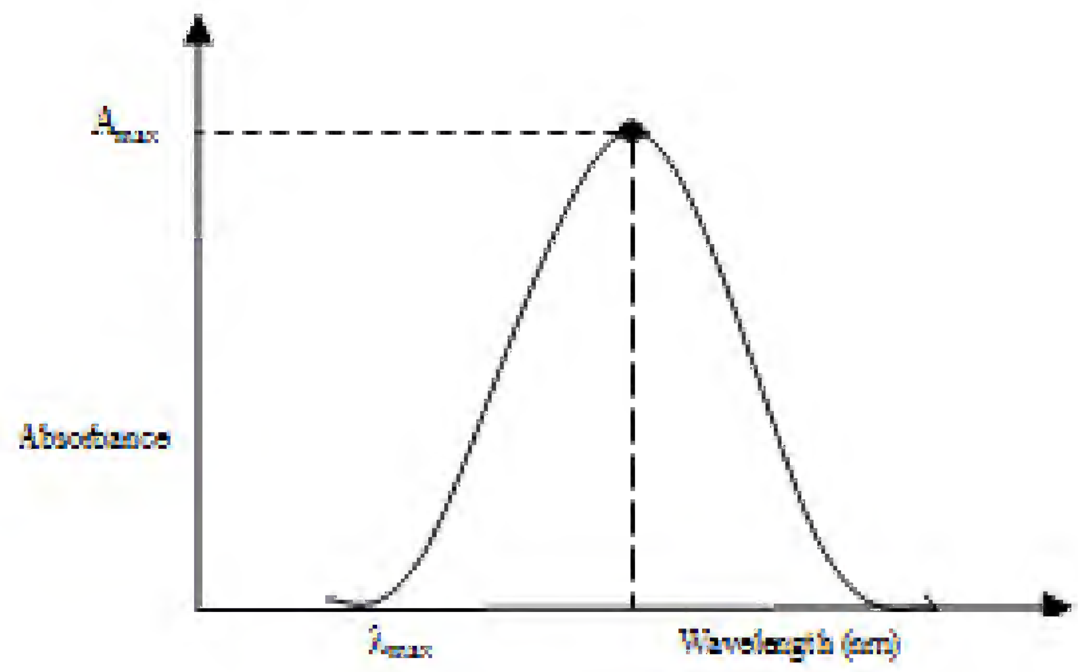

Figure 1: Graphical Analysis of Absorbance Data at Different Wavelengths

The relationship between the absorbance and concentration, given by equation (3), is known as Beer's Law [8]. Generally, the path length, L, is constant and it is generally equals to one cm. The equation (3) becomes,

$$A = \alpha C \tag{4}$$

For the particular chemical species, equation (4) can be compared with the equation of a straight line,

$$y = mx + b \tag{5}$$

Where, m is slope of the line, and b is the y-intercept. Comparison of equations (4) and (5) results into, $y = A$, $x = C$, $m = \alpha$, and $b = 0$. In ideal situation, if the concentration is zero then absorbance is also zero. It experimentally proves the Beer's law which states that a plot of absorbance verses concentration gives a straight line passing through the origin. Slope of the line depends on the constituents present in the solution, and it is required for the calculation of concentration of unknown species in the solution.

The value of absorbance cannot be used as concentration. The concentrations can be calculated from equation (4). Where, α can be obtained from the graph of concentration verses absorbance. This graph is called a calibration curve or Beer's plot [8] which is shown in Figure 2. The software program calculates the unknown concentration using the formula $C= A_{max}/\alpha$. Where α is the slope of curve shown in Figure 2 and A_{max} is the absorbance at λ_{max}.

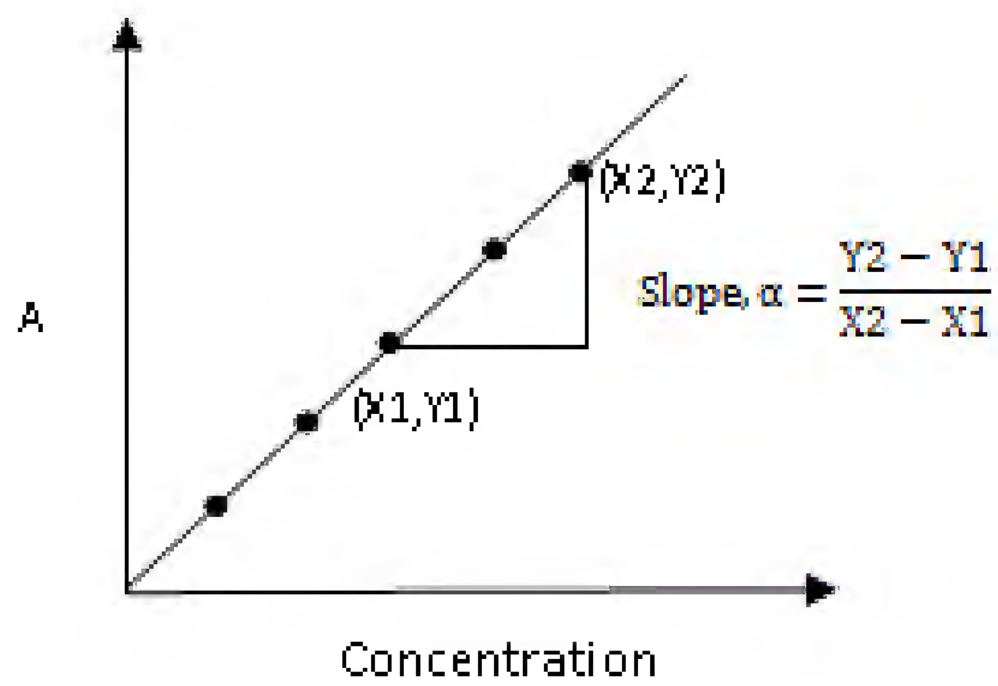

Figure 2: Plot of Concentration versus Absorbance (A)

3. CIRCUIT DIAGRAM AND DESCRIPTION

The block and circuit diagrams of the colorimeter are shown in Figure 3 and 4 respectively. The colorimeter mentioned in the present work consists of following main units: forward current control cum driver circuit for the LED, cuvette, photoconductive cell with dark current compensation circuit, signal processor with display, and dc power supply. Details of each of these units are given below:

3.1 Forward Bias Current Control and LED Driver Circuit

Software program generates constant frequency square wave with variable duty cycle. In other words ON-time of square wave can be changed by software program as per the requirement as shown in the flowchart depicted in the Figure 5. The square wave signal is applied to the constant current circuit. This is a very simple design using only 2 NPN transistors and 2 biasing resistors. The transistor T1 is used in the negative feedback loop of transistor T2. The negative feedback action maintains the LED current constant. This circuit is designed to use LEDs with barrier potential between 1.6 V and 6V with power consumption up to 10W. The transistor T1 can be medium power type like 2N2222, but transistor T2 must be power transistor such as 2N3055. LEDs with different colors used in the work are: Ultra-violet (365 nm), Pink (415), Blue (460 nm), Green (540nm), Yellow (590nm), Orange (620nm), Deep Red (700nm) and near-Infrared (750nm).

3.2 Cuvette

It is made from the water clear glass or quartz material and it should not absorb the light radiation. LED is fitted at one side of cuvette and its light is made to shine through the sample placed in cuvette. The transmitted light passed through the sample, struck the opposite side of the cuvette where the LDR is mounted [9].

3.3 Photoconductive Cell and Dark Current Compensation Circuit

Photoconductive cell is made from solid material with weakly bound valence electrons. If the surface of material is exposed to visible light then the electrons are excited to the conduction band and this phenomenon increases the bulk conductance of material [10]. The light dependant resistor (LDR) made from cadmium sulphide (CdS) is commonly used as photoconductivity cell. The resistance of LDR depends on the intensity and wavelength of the light falling on it. The resistance varies between 10 MΩ (dark) and 10 KΩ (sunlight) for a typical LDR. LDR is highly sensitive to the ambient light. The effect of ambient light and dark current can be minimized with use of appropriately designed resistance bridge. The resistors R_1, R_2, R_3 and R_{LDR} form the bridge. If the product R_1x R_3 equals to R_2 x R_{LDR} then the inputs to the difference amplifer are equal and its output is zero. The bridge can be balanced by varying the resistor R_3.

3.4 Signal Processor and Display Unit

The signal processing unit has the job of signal acquisition and manipulating it in the form which is required by the display system. The signal processing unit is a microcontroller system based on PIC18F4550 [11], which performs the operation of analog to digital conversion, data management and modification.

Signal Display and Keypad

The microcontroller has the function of analyzing and displaying data on the output device. It is a character type 16 x 2 LCD which accepts the command and the data in ASCII format. The LCD is interfaced to the port A of µC. The software program has been written to send the ASCII code in 4-bit mode and the user interface has been provided by the 4 X 4 keypad. It is constructed from the 'push to on release to off' type key switches. It is interfaced to port D of the microcontroller. The pressed key is detected by the program which scans keypad and calculates the key position with the use of switch statement of C-language.

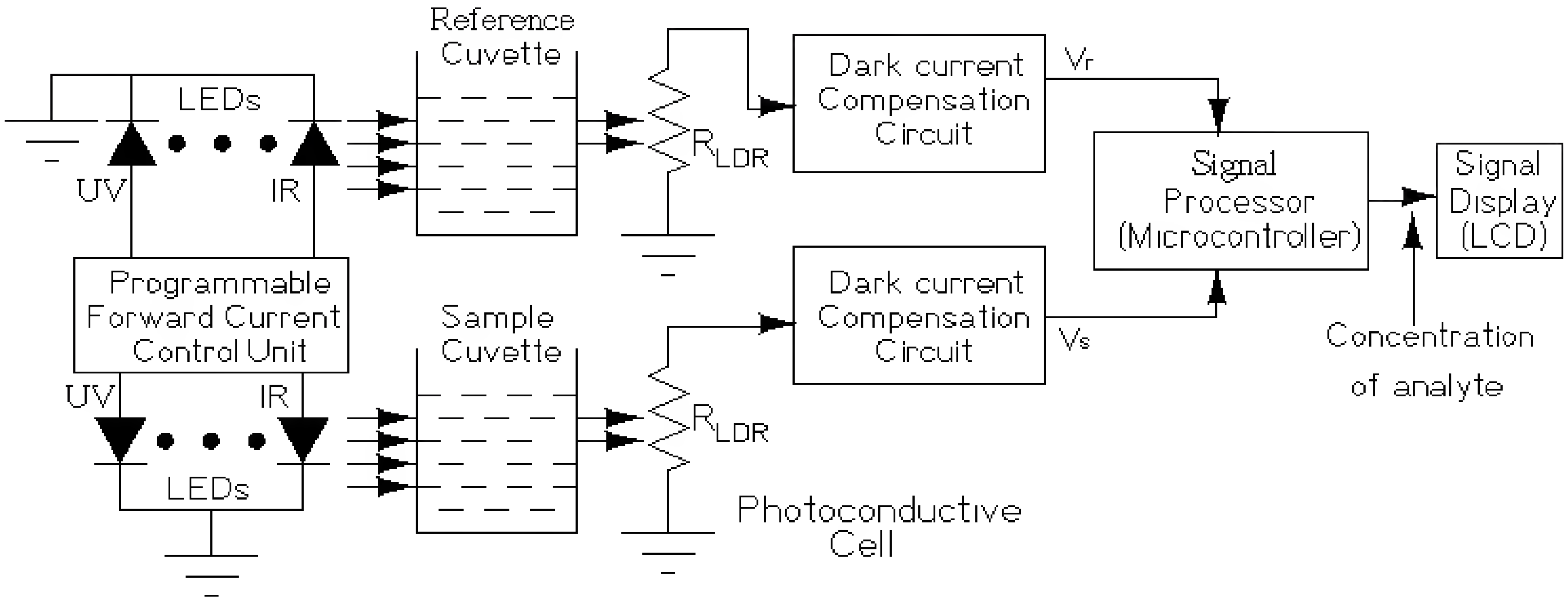

Figure 3: Block Diagram of Embedded System Based Colorimeter

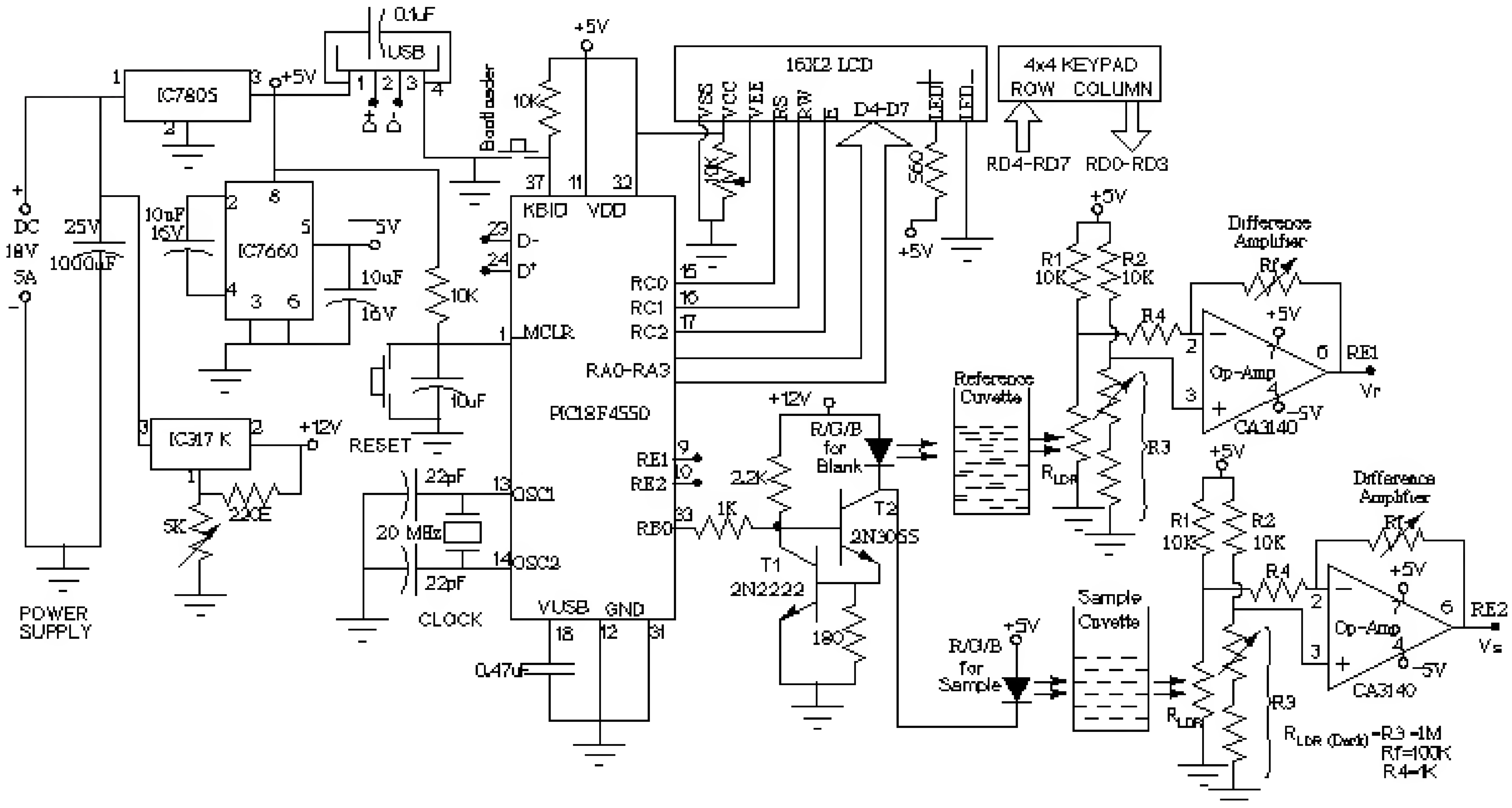

Figure 4: Circuit Diagram of Embedded System Based Colorimeter

4. SOFTWARE SECTION

The program is written in embedded C-language. It is edited in MPLAB IDE and compiled using C-18 compiler. The program is loaded into flash memory of PIC 18F4550 microcontroller with the use of Extreme burner. The various steps of the program development are shown in the flowchart as shown in Figure 5.

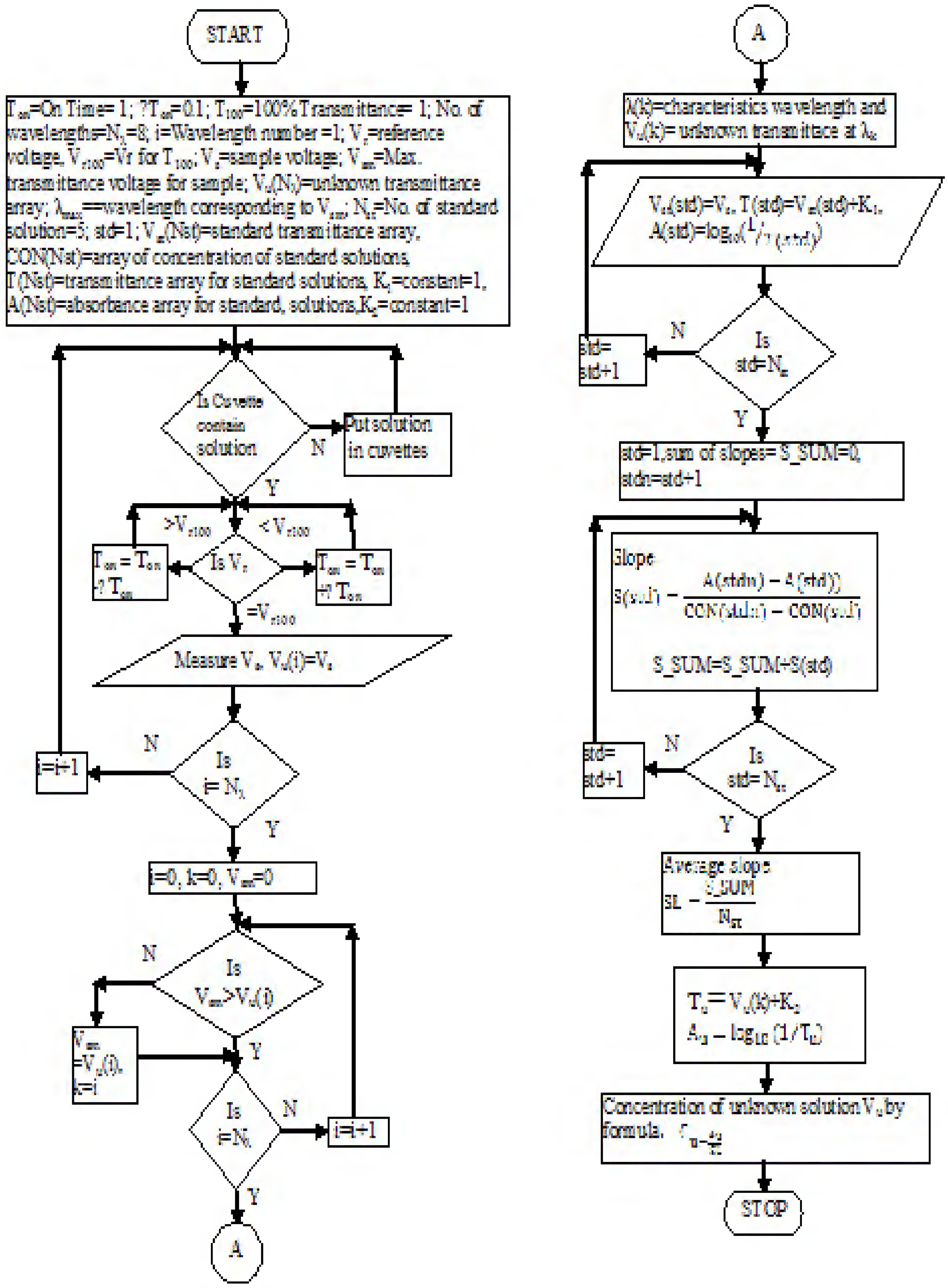

Figure 5: Flow Chart for the Embedded System Based Colorimeter

The program has following functions:

- To generate constant frequency square wave and modulate its on-time using pulse-width modulation logic for the adjustment of intensity of transmitted light at maximum (100%) for the reference solution.
- Measure the analog voltage, V_A, corresponding to the intensity of transmitted light in terms of digital value.
- To convert above digital value into transmittance. ($T = V_A + \text{const.}$)
- To scan the wavelength spectrum and find the characteristics wavelength i.e. λ_{max} and A_{max}.
- To find the type of chemical species.

- To convert transmittance into absorbance. ($A = \log_{10}(1/T)$).
- To find the absorption coefficient 'α' from the slope of calibration curve of concentration verses absorbance of the standard solutions. (α = slope).
- To find the concentration of unknown species. ($C_{un} = A_{max} / \alpha$).

5. RESULTS AND DISCUSSIONS

The concentration of unknown species is determined from the calibration curve. In order to draw the curve, the first step is to find the wavelength λ_{max} at which maximum absorbance obtained, and then the absorbance is measured for the series of known standard solutions [12]. The values of absorbance for unknown solution at different wavelengths are depicted in Table 1.

Table 1: Absorbance by Unknown Concentration Solution at Different Wavelengths

Wavelength, λ(nm)	Absorbance, A
400	0.22
420	0.17
470	0.16
500	0.07
530	0.06
620	0.35
660	0.62
700	0.72
750	0.57

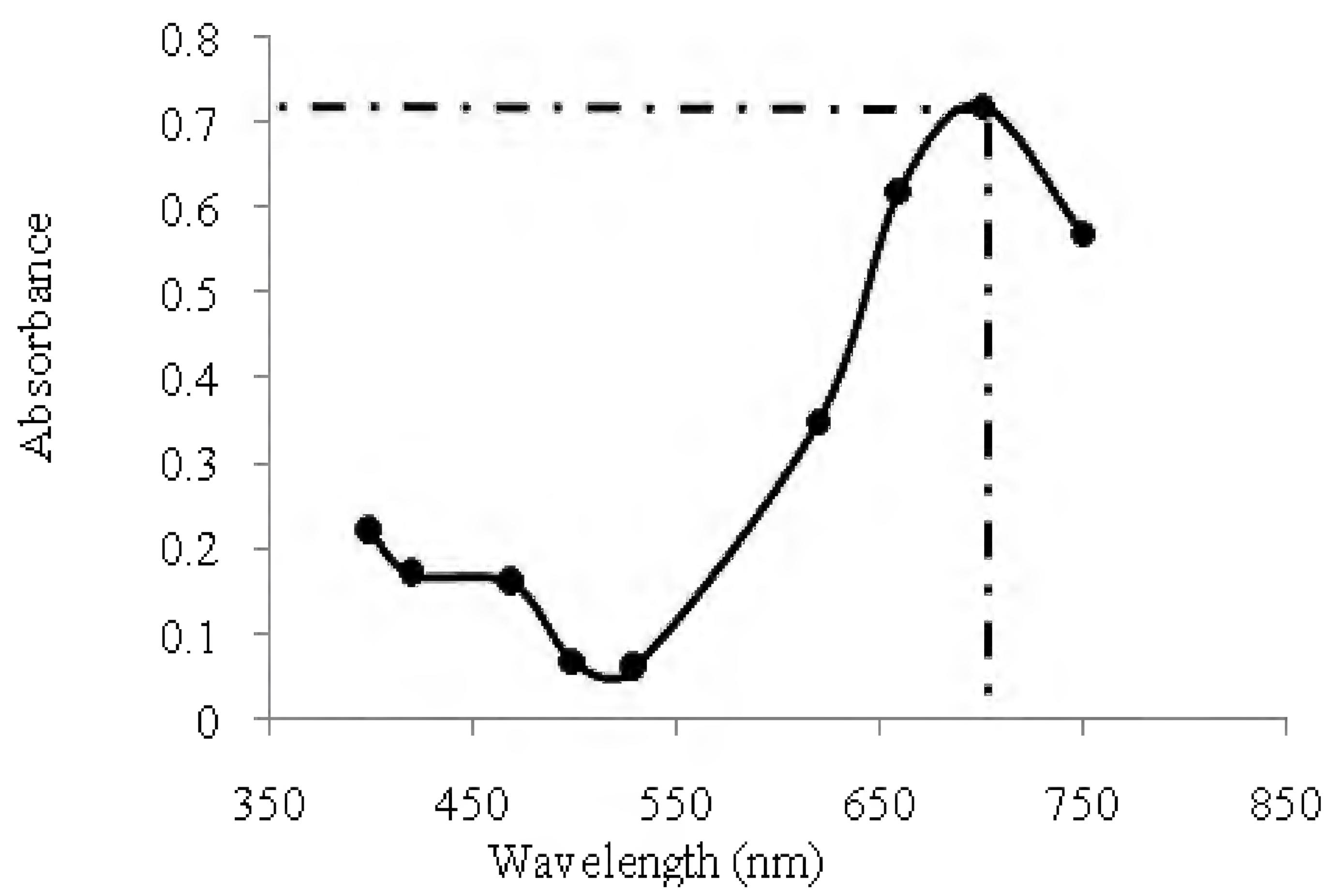

Figure 6: Wavelength vs Absorbance of Unknown Solution

The plot of wavelength versus absorbance is shown in Figure 6. It is revealed from Figure 6, that the maximum response is at 700 nm, which is the wavelength of infrared light. The absorbance of various concentrations of standard nickel and unknown amount of nickel in water are shown in Table 2. Five different concentrations of Ni^{2+} solutions were prepared. Using highest concentration solution of Ni^{2+}, λ_{max} was determined and it was found to be 700 nm. At this λ_{max}, absorbance of different concentration solutions of Ni^{2+} as well as unknown concentration solution of nickel were measured which are depicted in Table 2. The graph of concentration verses absorbance is shown in Figure 7.

Table 2: Absorbance for the Known and Unknown Concentrations of Nickel Solution at 700 nm

ml of 0.4 M NiSo4 Solution	Total Volume, ml	Concentration/ M	Absorbance
5	25	0.08	0.15
10	25	0.16	0.31
15	25	0.24	0.44
20	25	0.32	0.58
25	25	0.4	0.73
Unknown sample	**25**	**Unknown Concentration**	**0.18**

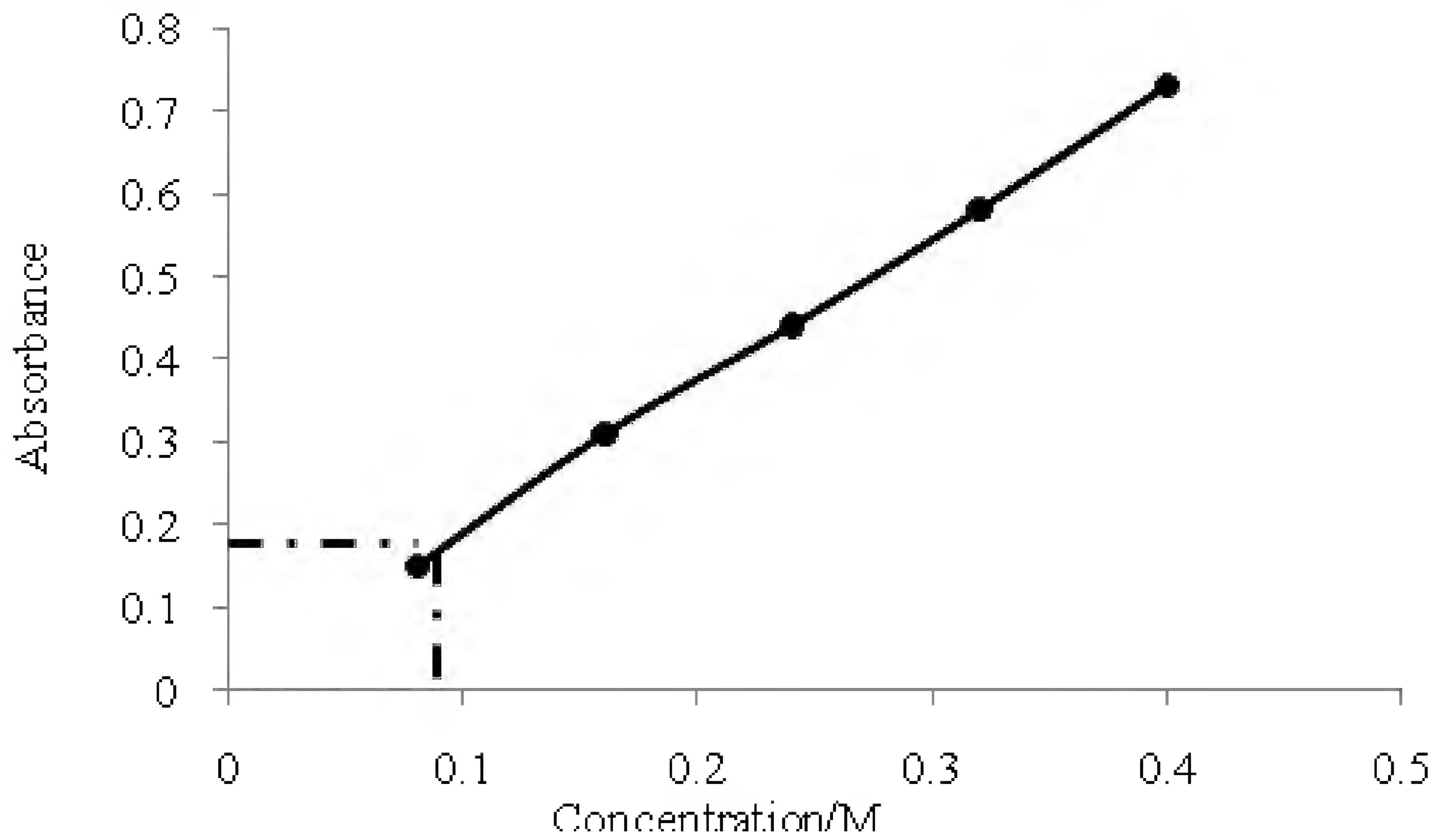

Figure 7: Calibration Curve for Finding the Unknown Concentration of Nickel in Water

For a given unknown solution the measured absorbance is 0.18. The concentration of unknown solution is determined from the calibration curve as shown in Figure 7. The value of unknown concentration of nickel from the curve is found to be 0.09 M.

6. CONCLUSIONS

Present work describes the prototype colorimeter with separate cuvettes for sample and blank solutions. The concentration of unknown sample is obtained with reference to the blank solution. Beer-Lambert law gives direct relation between absorption and concentration. But the reagent added in the colorless solution of nickel absorbs some part of incident light and introduces a constant error in the above relation. It is eliminated by use of experimentally determined calibration constant. The developed device is cheap, portable, and easy to operate. It has been successfully used for the detection of nickel in the waste water sample. The device can be used in the laboratory for the quantitative analysis of color solutions.

Limitations: LEDs are monochromatic and they are not available for all the colors. If the peaks response wavelength of LED is not equal to the characteristic wavelength of analyte then the instrument gives approximate results.

Future Scope: The developed colorimeter will be interfaced to the personal computer to perform the graphical analysis.

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